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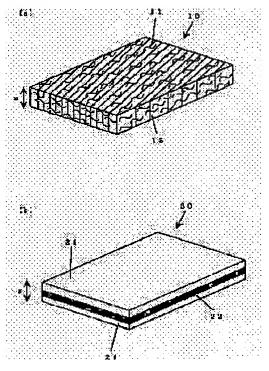
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(54) NONAQUEOUS ELECTROLYTE SECONDARY BATTERY

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a nonaqueous electrolyte secondary battery, having reduced internal resistance and superior high-temperature conservation and overcharging characteristics by preventing wrinkling of the separator, even if a polyvinylidene fluoride resin is used which has superior electrolyte sustainment as a separator material.

SOLUTION: This nonaqueous electrolyte secondary battery has a separator 10, formed with a composite resin film integrated by filling a reinforcing material layer 11 with a polyvinylidene fluoride resin 12 or a separator 20 formed with a composite resin film, in which a polyvinylidene fluoride resin layer 21 and a reinforcing material layer 22 are laminated. With the polyvinylidene



fluoride resin 12, filled with the reinforcing material layer 11 for integration or the polyvinylidene fluoride resin layer 21 laminated on the reinforcing material layer 22, the swelling of the polyvinylidene fluoride resin, if swollen, in the cross and longitudinal directions, is restricted by the reinforcing material 11 or 22, wrinkling of the separator 10 or 20 will not occur.

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- 3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[Field of the Invention] This invention relates to the nonaqueous electrolyte rechargeable battery equipped with the positive electrode which uses as positive active material occlusion, the negative electrode from which it may be desorbed, and a lithium content metallic oxide for a lithium ion, nonaqueous electrolyte, and the separator which isolates a positive electrode and a negative electrode. [0002]

[Description of the Prior Art] In recent years, the miniaturization of electronic equipment and lightweight-izing are remarkable, and its request of the formation of small lightweight is very large also to the cell which serves as a power source in connection with it. Then, the nonaqueous electrolyte rechargeable battery which is a small light weight and is represented with high capacity with a lithium ion battery as a cell in which charge and discharge are possible comes to be put in practical use, and it came to be used for a portable electron, communication equipment, etc., such as a small video camera, a cellular phone, and a notebook computer.

[0003] Occlusion and the carbon system ingredient from which it may be desorbed are used for this kind of nonaqueous electrolyte rechargeable battery for a lithium ion as a negative-electrode active material. The lithium content transition-metals oxide of LiCoO2, LiNiO2, LiMn 2O4, and LiFeO2 grade is used as positive active material. After assembling as a cell using the electrolytic solution which dissolved lithium salt in the organic solvent as a solute, it is the cell by which the lithium ion which came out of positive active material by first-time charge enters in a carbon particle, and the charge and discharge of it become possible.

[0004]

[Problem(s) to be Solved by the Invention] By the way, if it is in such a nonaqueous electrolyte rechargeable battery, in order to use nonaqueous electrolyte, fine porosity film, such as polyolefine system resin with it, for example, polyethylene, (PE), and polypropylene (PP), has been used. [low and reactivity with an organic solvent and] [cheap as a separator which isolates a positive electrode and a negative electrode] However, fine porosity film, such as polyethylene and polypropylene, had the problem that the solution retention of the electrolytic solution was low, in order to hold the electrolytic solution in the hole part of the fine porosity film. And when the solution retention of the electrolytic solution was low, cell internal resistance increased, and while the cycle property fell, there was a problem that a overcharge property also fell.

[0005] For this reason, although it came to use nonwoven fabrics, such as polypropylene (PP) and polyethylene terephthalate (PET), as a separator in order to improve the solution retention of the electrolytic solution, nonwoven fabrics, such as polypropylene (PP) and polyethylene terephthalate (PET), had the problem that a cycle property fell, like the fine porosity film. Then, it came to be proposed that solution retention uses the good polyvinylidene fluoride resin film as a separator. This polyvinylidene fluoride resin film has the good holdout of the electrolytic solution, since adhesion with an electrode is possible, cell internal resistance falls and its cell property improves.

[0006] However, while the polyvinylidene fluoride resin film was rich in solution retention, it produced the problem that a dimensional change was large, by swelling. Namely, if the separator 60 which consists of polyvinylidene fluoride resin film holds the electrolytic solution as shown in <u>drawing 5</u>, it will swell in all the directions of the cross direction (the x directions of <u>drawing 5</u>) of a separator 60, the die-length direction (the direction of y of <u>drawing 5</u>), and the height direction (the direction of z of <u>drawing 5</u>).

[0007] By the way, as shown in <u>drawing 6</u>, this kind of nonaqueous electrolyte rechargeable battery usually A positive electrode 70 (in addition, the positive-active-material layer 72 is applied by both sides of an axis 71, and this positive electrode 70 is formed in them), and a negative electrode 80 (in addition) Although this negative electrode 80 makes a separator 60 intervene, carries out a laminating, wind it spirally, it is made into an electrode group, inserts this into a cell case and is manufactured while the negative-electrode active material layer 82 is applied and formed in both sides of an axis 81 When the separator 60 swelled, the wrinkling arose in the separator 60, the space sections 61 and 62 arose between the separator 60, the positive electrode 70, or the negative electrode 80, and the problem that internal resistance increased was produced.

[0008] Moreover, if a wrinkling arose in a separator 60 and the space sections 61 and 62 arose between a separator 60, a positive electrode 70, or a negative electrode 80, since the reaction in the space sections 61 and 62 would become an ununiformity, when it becomes easy to generate a dendrite and the dendrite occurred, the problem that an internal short circuit occurred was also produced. Then, it is made in order that this invention may solve the above-mentioned trouble, and even if it uses polyvinylidene fluoride resin excellent in the solution retention of the electrolytic solution as a separator ingredient, do not produce a wrinkling, it is made a separator and internal resistance is reduced, and it aims at offering the nonaqueous electrolyte rechargeable battery excellent in the cycle property and the overcharge property.

[The means for solving a technical problem, and its operation and effectiveness] In order to attain such a purpose, he is trying to form the nonaqueous electrolyte rechargeable battery of this invention by the compound resin film which was filled up with polyvinylidene fluoride resin and unified in the compound resin film which carried out the laminating of a polyvinylidene fluoride resin layer and the reinforcing materials layer for the separator, or a reinforcing materials layer. Thus, if the laminating of the polyvinylidene fluoride resin layer is carried out in a reinforcing materials layer, or it is filled up with polyvinylidene fluoride resin and unifies in a reinforcing materials layer, even if polyvinylidene fluoride resin holds and swells the electrolytic solution, since the swelling to the cross direction and the die-length direction is regulated by reinforcing materials, it will not produce a wrinkling in a separator. For this reason, internal resistance falls and the nonaqueous electrolyte rechargeable battery excellent in the cycle property and the overcharge property comes to be obtained.

[0010] In this case, although it is necessary to choose from the quality of the material which is not swollen even if it holds the electrolytic solution as a reinforcing materials layer, since the nonwoven fabric made from polyethylene terephthalate, the nonwoven fabric made from polypropylene, the nonwoven fabric made from polyethylene, the fine porosity film made from polypropylene, etc. have the property in which it does not swell even if it holds the electrolytic solution, they are desirable.

[0011] And if thickness of a reinforcing materials layer is thickened, even if the mechanical strength of a separator will increase and a polyvinylidene fluoride resin layer will swell, producing a wrinkling in a separator is lost, but since the inside of a cell is the limited space, if thickness of a reinforcing materials layer is thickened, only the part will need to make thickness of a polyvinylidene fluoride resin layer thin, and the holdout of the electrolytic solution will fall. For this reason, even if it sets thickness of a reinforcing materials layer to 30 micrometers or less and a polyvinylidene fluoride resin layer swells it, it is desirable to consider as the thickness of extent which does not produce a wrinkling in a separator. [0012] Furthermore, it is desirable to use one or more sorts chosen from 6 vinylidene fluoride homopolymer or vinylidene fluoride, 3 fluoride-salt-ized ethylene and tetrafluoroethylene, and propylene fluoride and ethylene as polyvinylidene fluoride of vinylidene fluoride copolymers.

[0013]

above.

[Embodiment of the Invention] Below, 1 operation gestalt of the nonaqueous electrolyte rechargeable battery of this invention is explained based on <u>drawing 1</u> thru/or <u>drawing 3</u>. In addition, <u>drawing 1</u> is the perspective view showing the separator of this invention typically, <u>drawing 1</u> (a) shows the compound resin film which was filled up with polyvinylidene fluoride resin and unified in the reinforcing materials layer, and <u>drawing 1</u> (b) shows the compound resin film which carried out the laminating of a polyvinylidene fluoride resin layer and the reinforcing materials layer. <u>Drawing 2</u> is the sectional view showing the condition of having carried out these separators in between and having carried out the laminating of a positive-electrode plate and the negative-electrode plate, <u>drawing 2</u> (a) is the sectional view showing the condition of having carried out the laminating using the separator of <u>drawing 1</u> (a), and <u>drawing 2</u> (b) is the sectional view showing the condition of having carried out the laminating using the separator of <u>drawing 1</u> R> 1 (b). <u>Drawing 3</u> is drawing showing typically the nonaqueous electrolyte rechargeable battery which these layered products were ****(ed), and was contained and formed in the lamination sheathing inside of the body.

- [0014] 1. In this nonwoven fabric 11 made from polyethylene terephthalate (PET), it was filled up with polyvinylidene fluoride (PVdF) resin 12, and unified, using the nonwoven fabric 11 made from polyethylene terephthalate (PET) (thickness of 30 micrometers) as production (1) example 1 reinforcing materials of a separator, and the separator 10 which consists of compound resin film was produced. This separator 10 was used as the compound resin film a of an example 1.
- [0015] (2) In this nonwoven fabric 11 made from polypropylene (PP), it was filled up with polyvinylidene fluoride (PVdF) resin 12, and unified, using the nonwoven fabric 11 made from polypropylene (PP) (thickness of 30 micrometers) as example 2 reinforcing materials, and the separator 10 which consists of compound resin film was produced. This separator 10 was used as the compound resin film b of an example 2.
- [0016] (3) The laminating of the polyvinylidene fluoride (PVdF) resin film 21 and 21 was pasted up and carried out to both sides of the fine porosity film 22 made from this the (polyethylene PE), using the fine porosity film (thickness of 30 micrometers) 22 made from (Polyethylene PE) as example 3 reinforcing materials, and the separator 20 which consists of compound resin film was produced. This separator 20 was used as the compound resin film c of an example 3.
- [0017] (4) The separator 60 (refer to <u>drawing 5</u>) was produced only using the polyvinylidene fluoride (PVdF) resin film (thickness of 30 micrometers), without using the example reinforcing materials of a comparison. This separator 60 was used as the resin film x of the example of a comparison. [0018] In addition, what is necessary is just to use one or more sorts chosen from 6 vinylidene fluoride homopolymer or vinylidene fluoride, 3 fluoride-salt-ized ethylene and tetrafluoroethylene, and propylene fluoride and ethylene of vinylidene fluoride copolymers as polyvinylidene fluoride mentioned
- [0019] 2. The positive active material which consists of lithium content diacid-ized cobalt (LiCoO2) heat-treated at the temperature of 700-900 degrees C of production of a positive-electrode plate, the graphite as an electric conduction agent and KETCHIEN black, and the fluororesin as a binder were mixed at a rate of 90:3:2:5 with the mass ratio, this was dissolved in the organic solvent which consists of a N-methyl-2-pyrrolidone (NMP), and it considered as the paste 32. This paste 32 was applied to homogeneity with the doctor blade method etc. to both sides of the metal axis (for example, aluminium foil whose thickness is 20 micrometers) 31. Subsequently, after having passed the inside of the heated dryer, carrying out vacuum heat treatment at the temperature of 100-150 degrees C and removing the organic solvent which was required at the time of paste production, it rolled out with the roll press machine, and the positive-electrode plate 30 was produced so that thickness might be set to 0.17mm. [0020] 3. The negative-electrode active material which consists of production one side of a negative-electrode plate and a natural graphite (d= 3.36A), and the fluororesin as a binder were mixed at a rate of 95:5 with the mass ratio, this was dissolved in the organic solvent which consists of a N-methyl-2-pyrrolidone (NMP), and it considered as the paste 42. This paste 42 was applied to homogeneity over the whole surface of both sides of the metal axis (for example, copper foil whose thickness is 20

micrometers) 41 with the doctor blade method etc. Subsequently, after having passed the inside of the heated dryer, carrying out vacuum heat treatment at the temperature of 100-150 degrees C and removing the organic solvent which was required at the time of paste production, it rolled out with the roll press machine, and the negative-electrode plate 40 was produced so that thickness might be set to 0.14mm. [0021] 4. After attaching positive-electrode current collection tab 31a in the axis 31 of the positive-electrode plate 30 which is the production **** of a nonaqueous electrolyte rechargeable battery, and was made and produced and attaching negative-electrode current collection tab 41a in the axis 41 of the negative-electrode plate 40, the laminating of the separators 10, 20, and 60 which consist of each resin film a, b, and cx produced as mentioned above was carried out and carried out in between, respectively. Then, after having ****(ed) by the winder which is not illustrated, carrying out the tape stop of the outermost periphery and considering as a spiral electrode object, this spiral electrode object was crushed flatly and it considered as the plate. Subsequently, to the mixed solvent which mixed ethylene carbonate (EC) and diethyl carbonate (DEC) so that a volume ratio might be set to 3:7, as an electrolyte salt, 1., 0.05 mols /, and LiN (SO2C2F5)2 dissolved 1 in 0.95 mols /, and LiPF6 prepared the electrolytic solution.

[0022] Subsequently, superposition and the edges of these pairs were heat sealed for the edge of the laminate material of 5 layer structures which contain from an outside the adhesives layer which becomes order from polyethylene terephthalate (PET), adhesives, aluminum, adhesives, and polypropylene, the obturation sections 53 and 54 were formed, and the tubed sheathing object 50 was produced. Subsequently, as shown in <u>drawing 3</u>, as it projected from one opening of the sheathing object 50, the plate which crushed the spiral electrode object flatly and formed it was inserted in the sheathing inside of the body, and where positive-electrode current collection tab 31a and negative-electrode current collection tab 41a are inserted into opening of one of these, positive-electrode current collection tab 31a and negative-electrode current collection tab 41a heat sealed this opening, and formed the obturation section 51.

[0023] Subsequently, after pouring in the electrolytic solution prepared as mentioned above from opening of another side of this sheathing object, this opening was heat sealed, the obturation section 52 was formed and sealed, and four kinds of nonaqueous electrolyte rechargeable batteries A, B, C, and X were produced, respectively. Thus, the capacity of each produced nonaqueous electrolyte rechargeable batteries A, B, C, and X was 500mAh(s). In addition, the nonaqueous electrolyte rechargeable battery produced using the compound resin film a of an example 1 was used as Cell A, the nonaqueous electrolyte rechargeable battery produced using the compound resin film b of an example 2 was used as Cell B, the nonaqueous electrolyte rechargeable battery produced using the compound resin film c of an example 3 was used as Cell C, and the nonaqueous electrolyte rechargeable battery produced using the resin film x of the example of a comparison was used as Cell X.

[0024] 5. When the alternating current whose frequency is 1kHz was impressed between forward and the negative-electrode terminal of each of these cells A, B, C, and X and the internal impedance (momega/cm2) of each cells A, B, C, and X was measured using each cells A, B, C, and X which are the measurement ****s of an impedance, and were made and produced, a result as shown in the following table 1 was brought.

[0025]

[Table 1]

電池種類	セパレータの 種 類	インピーダンス (mΩ/cm²)
Α	複合樹脂膜a	0.18
В	複合樹脂膜b	0.19
С	複合樹脂膜c	0.20
Х	樹脂膜x	0.26

[0026] The cell A using the compound resin film a (thing using the nonwoven fabric made from polyethylene terephthalate (PET) as reinforcing materials) so that clearly from the above-mentioned table 1 The cell B using the compound resin film b (thing using the nonwoven fabric made from polypropylene (PP) as reinforcing materials), and the cell C using the compound resin film c (thing using the fine porosity film made from (Polyethylene PE) as reinforcing materials) It turns out that internal impedance is lower than the cell X using the resin film x (thing using only the polyvinylidene fluoride (PVdF) resin film without using reinforcing materials).

[0027] While the polyvinylidene fluoride (PVdF) resin film has good solution retention, by absorbing the electrolytic solution, it swells in all the directions of the cross direction, the die-length direction, and the thickness direction, a wrinkling arises, the space section arises between a positive electrode 30 or a negative electrode 40, and since internal resistance (internal impedance) increased, this is considered. On the other hand, fill up with polyvinylidene fluoride (PVdF) resin the reinforcing materials who consist of a nonwoven fabric made from polypthylene terephthalate (PET), and a nonwoven fabric made from polypropylene (PP), and unify, or Or if the laminating of the polyvinylidene fluoride (PVdF) resin film is carried out by the reinforcing materials who consist of fine porosity film made from (Polyethylene PE), even if it swells with the electrolytic solution Since it is regulated by reinforcing materials and the swelling direction turns into only the thickness direction (the direction of z of drawing 1), a wrinkling does not arise in separators 10 and 20 and the swelling of the cross direction and the dielength direction is considered that internal resistance (internal impedance) fell.

[0028] 6. a cycle trial -- using the occasion and the nonaqueous electrolyte rechargeable batteries A and X produced as mentioned above, constant-current charge was carried out until cell voltage was set to 4.1V by the 500mA (1C) charging current, and after that, it charged by the constant voltage of 4.1V for 3 hours, and considered as the full charge condition. Then, a result of the discharge capacity for each [as opposed to / perform the charge-and-discharge cycle trial of making it discharge until a termination electrical potential difference is set to 2.75V according to a 500mA (1C) discharge current at the room temperature after making it stop for 10 minutes at a room temperature, and / initial discharge capacity] cycle of every when it asks for (%) comparatively and expresses with a graph as a cycle property, as shown in drawing 4 was brought.

[0029] It turns out that the cycle property of the cell A using the compound resin film a (thing using the nonwoven fabric made from polyethylene terephthalate (PET) as reinforcing materials) is improving from the cell X which used the resin film x (thing using only the polyvinylidene fluoride (PVdF) resin film without using reinforcing materials) so that clearly from drawing 4. If this was in Cell X, since only the polyvinylidene fluoride (PVdF) resin film was used as a separator, it swells in all the directions of the cross direction, the die-length direction, and the thickness direction, and a wrinkling arises, and since internal resistance increased and the cycle property fell, this separator is considered.

[0030] 7. a continuation overcharge trial -- capacity (2000mAh) 4 times quantity of electricity of a cell performed continuation constant-current charge by the 500mA (1C) charging current using the occasion and the nonaqueous electrolyte rechargeable batteries A, B, and X produced as mentioned above, and when asked for the rate (%) which abnormalities, such as an internal short circuit, generated, a result as shown in the following table 2 was brought.

[0031] [Table 2]

電池種類	セパレータの 種 類	異常の発生割合 (%)	異常の状態
A	複合樹脂膜 a	0	異常なし
В	複合樹脂膜 b	0	異常なし
х	樹脂膜x	100	内部短絡

[0032] If it is in the cell X using the resin film x (thing using only the polyvinylidene fluoride (PVdF) resin film without using reinforcing materials) so that clearly from the above-mentioned table 2 As opposed to abnormalities called an internal short circuit having arisen Abnormalities all did not produce the cell A using the compound resin film a (thing using the nonwoven fabric made from polyethylene terephthalate (PET) as reinforcing materials), and the cell B using the compound resin film b (thing using the nonwoven fabric made from polypropylene (PP) as reinforcing materials).

[0033] If this was in Cell X, since only the polyvinylidene fluoride (PVdF) resin film was used as a separator, this separator is swollen in all the directions of the cross direction, the die-length direction, and the thickness direction, a wrinkling produces it, the space section arises between a separator, a positive electrode, or a negative electrode, and it is considered that the short circuit arose owing to that the dendrite occurred in this space section.

[0034] Since he is trying to form the compound resin film or the polyvinylidene fluoride resin layer 21 which was filled up with polyvinylidene fluoride resin 12 into the reinforcing materials layer 11, and unified separators 10 and 20, and the reinforcing materials layer 22 in this invention by the compound resin film which carried out the laminating as mentioned above Even if polyvinylidene fluoride resin 12 or the polyvinylidene fluoride resin layer 21 holds and swells the electrolytic solution, since the swelling to the cross direction and the die-length direction is regulated by reinforcing materials 11 and 22, it does not produce a wrinkling in separators 10 and 20. For this reason, internal resistance falls and the nonaqueous electrolyte rechargeable battery excellent in the cycle property and the overcharge property comes to be obtained.

[0035] And although producing a wrinkling in a separator 20 is lost even if the mechanical strength of a separator 20 will increase and the polyvinylidene fluoride resin layer 21 will swell, if thickness of the reinforcing materials layer 22 is thickened when carrying out the laminating of the polyvinylidene fluoride resin layer 21 to the reinforcing materials layer 22 Since the inside of a cell is the limited space, if thickness of the reinforcing materials layer 22 is thickened, only the part will need to make thin thickness of the polyvinylidene fluoride resin layer 21, and the holdout of the electrolytic solution will fall. For this reason, even if it sets thickness of the reinforcing materials layer 22 to 30 micrometers or less and the polyvinylidene fluoride resin layer 21 swells it, it is desirable to consider as the thickness of extent which does not produce a wrinkling in a separator 20.

[0036] In addition, in an above-mentioned operation gestalt, although the example using the laminate material of 5 layer structure as a sheathing object was explained, a sheathing object may use the product made from stainless steel, or the metal sheathing can made from aluminum in addition to the above-mentioned laminate material. Moreover, although the example using a natural graphite (d= 3.36A) as a negative-electrode active material was explained, occlusion, the metallic oxides (SnO, SiO2, etc.) from which it may be desorbed, the intermetallic compound (Li-aluminum) of a lithium, etc. are [lithium ion] suitable [in occlusion and the carbon system ingredient from which it may be desorbed, for example, carbon black, corks glassy carbon, carbon fibers, these baking objects or a lithium ion] in an above-mentioned operation gestalt, in addition to a natural graphite.

[0037] Moreover, in an above-mentioned operation gestalt, although the example using LiCoO2 as

positive active material was explained The lithium content transition-metals compound which can accept a lithium ion as a guest in addition to LiCoO2, For example, although LiNiO2, LiCoXNi(1-X) O2, LiCrO2, LiVO2, LiMnO2, LiFeO2, LiTiO2, LiScO2 and LiYO2, and LiMn2O4 grade are desirable It is suitable to use LiMn2O4, LiCoO2, and LiCoXNi(1-X) O2 independently, or to mix and use these two or more sorts especially.

[0038] Furthermore, as the electrolytic solution, it is the ion conductor which dissolved lithium salt in the organic solvent as a solute, and it can be used, if ionic conductivity is high, it is electrochemically [chemically and] stable to each forward and negative electrode, and an usable temperature requirement is wide and it is [safety is high and] cheap. For example, these mixed solvents, such as propylene carbonate (PC), sulfolane (SL), a tetrahydro furan (THF), and gamma-butyrolactone (GBL), are suitable in addition to the above-mentioned organic solvent.

[0039] Moreover, you may make it use the gel electrolyte containing polymerization nature compounds, such as an acrylate system polymer and an urethane acrylate system polymer. furthermore, except for LiPF [which used and described above the strong lithium salt of electronic suction nature as a solute]6, or LiN(SO2C2F5)2 -- LiBF4, LiClO4, LiAsF6, and LiSO3 -- CF3, LiN (SO2CF3)2, and LiSO3C4F9 grade are suitable. [for example,]

[Translation done.]

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